CHAPTER ONE

INTRODUCTION

σ

Initial State

Final State
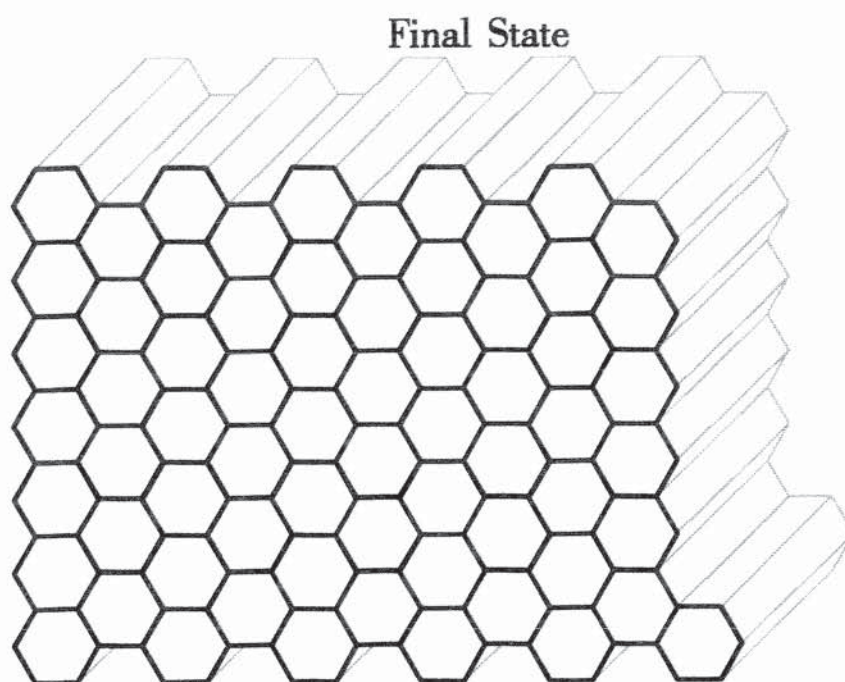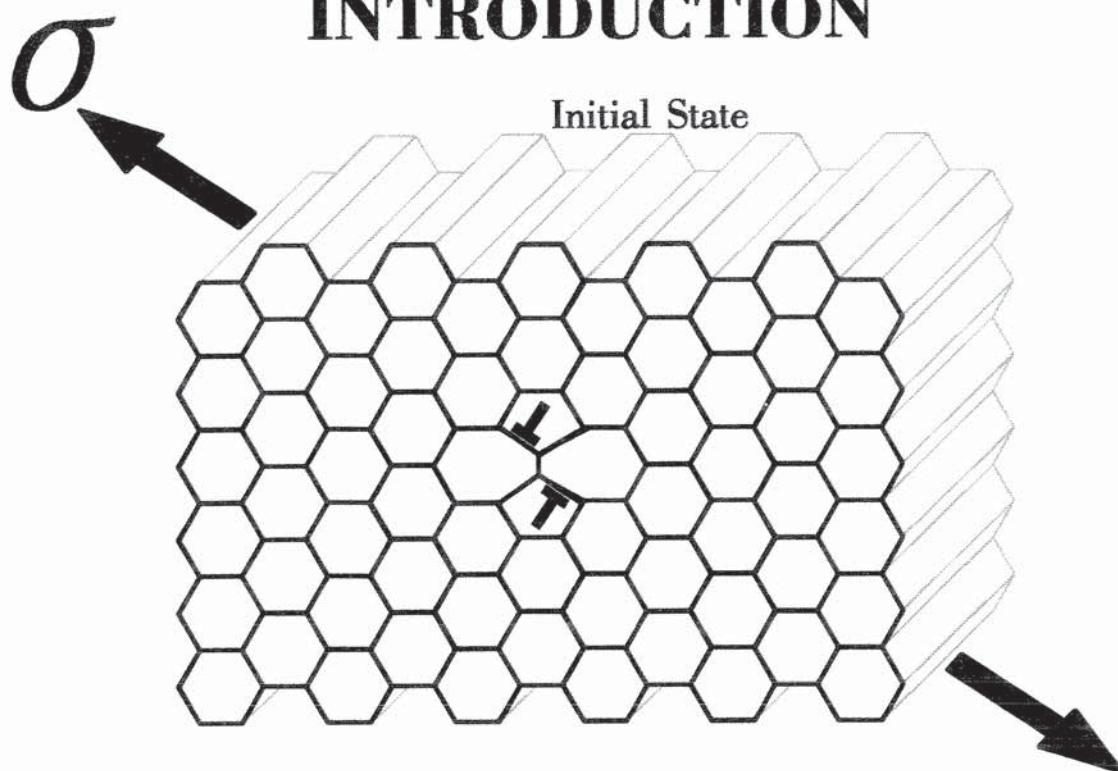
Superplasticity is a *behavior* exhibited by fine-grained polycrystalline materials subjected to low stresses, and temperatures in excess of approximately one-half their absolute melting point: a large, uniformly distributed strain is developed relatively quickly with minimal microstructural change. It is possible that superplasticity results from processes similar to those responsible for "conventional" creep: for both superplastic and diffusional creep deformation the flow stress increases with the imposed strain rate, and the flow stress is also higher at a given strain rate if the grain size is larger. Creep processes may not, however, be generally rate limiting during superplastic deformation because the strain rates predicted from models of these processes are usually several orders of magnitude smaller than the strain rates observed in superplasticity (Edington *et al.* 1976). Perhaps then, unique mechanisms lead to superplastic flow; such mechanisms might occur along with "conventional" creep mechanisms or even supplant them. As strain rates and deformation temperatures change so do the mechanisms which contribute to superplastic flow. Dislocation creep replaces superplastic flow if the strain rate is raised too much; at very low strain rates diffusional creep occurs instead of superplastic flow.

Polycrystalline materials suffer creep deformation upon exposure to low stresses and high temperatures for long periods of time. Their microstructures are therefore provided the opportunity to assume thermodynamically more stable states through phase transformations and coarsening (Cottrell 1953). Of particular interest in superplasticity is the coarsening of the grains (crystallites) comprising the polycrystal. This *grain growth* results when small grains disappear and adjacent grains enlarge to fill the vacated space (Burke and Turnbull 1952, Atkinson 1988). Often the rate of grain growth during superplastic deformation is

found to be substantially higher than the grain growth rate expected from a comparable annealing treatment (Kashyap *et al.* 1985), *i.e. deformation-enhanced grain growth* (Clark and Alden 1973) is observed.

Molten glass exhibits superplastic behavior when very narrow fibers are drawn from a thick rod, Figure 1a. This substance is *Newtonian viscous* since the fiber moves at a velocity, v, proportional to the applied force, F: $F = Bv$, where B is a constant. The fibers resist *necking, i.e.* they have little, if any tendency to pinch down locally, so that the applied force acts over the entire fiber length. The force divided by the specimen cross-sectional area is the stress, or *flow stress*, since the material flows with the force. Most metal alloys on the other hand will tend to neck when a tensile force is applied, as shown by Figure 1b. Then the applied force produces much greater stresses in the neck, concentrating plastic deformation there, which leads to relatively rapid failure (separation of the specimen into two pieces). The flow stress of a superplastic alloy is very "sensitive" to the strain rate: While not always linear like Newtonian materials, the flow stress will nevertheless increase markedly with the strain rate. An incipient neck—straining faster than the surrounding material—will therefore have a higher flow stress than its surroundings; deformation consequently tends to spread away from any small neck to the softer material around it. Superplastic materials therefore have a natural tendency to *"strain harden"* so that only *diffuse necks* (Figure 1b) occur, which makes very large strains a possibility.

As illustrated by Figure 1c, the *homogeneous grain growth* associated with the material's tendency to coarsen at the deformation temperature is generally considered to be deleterious to the diffuse necking process. Such *"static" grain growth* occurs throughout the

tensile specimen and therefore does not enhance ductility through "strain hardening:" the flow stress throughout the tensile specimen is likely to be increased. Deformation-enhanced grain growth is inhomogeneous; it only occurs where the specimen is deforming, and the rate of this coarsening increases with the strain rate, so it can cause "strain hardening:" Deformation-enhanced grain growth may "stabilize" an incipient neck by locally increasing the flow stress, so that the strain rate in surrounding, softer regions is increased, thereby "diffusing" the neck. The effects of "static" and deformation-enhanced grain growth on superplastic behavior are both pronounced and subtle: Relatively small changes (much less than a factor of ten) in grain size can produce either beneficial or deleterious effects, depending on the strain rate, strain rate path, and grain growth kinetics; see Hamilton (1989) for further details. **Accurate formulations of both "static" and deformation-enhanced grain growth kinetics therefore benefits selection of optimal forming procedures.**

Figure 1d shows the three strain rate regimes commonly exhibited by superplastic metal alloys: Region I, Region II, and Region III, from low to high strain rates. The strain rate sensitivity is largest in *Region II*; diffusional creep is exhibited for strain rates lower than those of *Region I*; *Region III* is dislocation creep. See Sherby and Wadsworth (1989) for discussion of a broader range of material behavior which may be considered superplastic.

Superplastic metal alloys are always *polycrystalline*: their microstructures consist of very small crystallites, or grains, as shown in two dimensions by Figure 1c, and in three dimensions by Figure 2. Interfaces between the crystallites in a polycrystalline material are called *grain boundaries*. Figure 3 shows a simple example of the atomic arrangements at a grain boundary; it is a *simple tilt boundary* in a lattice with six*fold* symmetry.

Grain boundaries are described as a honeycomb, or *hexagonal array*, in a two dimensional model of a polycrystalline material. Grain boundary structure and the lattices between boundaries are not detailed in such a model; the array of cells is therefore a *foam* or *froth*. Cell corners (*triple points*) in a two dimensional froth are *trivalent*, three boundaries emerge from a single corner. If all cells are uniform hexagons, then space is partitioned with the <u>minimum amount of boundary length</u> (Kappraff 1991). This is an important consideration: atomic bonds are incomplete in a grain boundary, since the atoms there do not belong to either grain, so there is a tendency ("driving force") for the atoms in these regions to join one of the adjacent grains where complete atomic bonding is possible.

Grain boundaries, like dislocations, contribute relatively little entropy to the crystal matrix (Nabarro 1967), consequently increase the free energy of the crystal, and since they are planar, have an associated energy per unit area, or *surface tension*. Surface tension is the reason why grain boundaries are modelled as in Figures 2 and 3. The decrease in free energy associated with the elimination of grain boundaries from the microstructure is the "driving force" for grain growth (Hillert 1965).

The average grain size (*d*) of a polycrystalline material increases when it is annealed by $d \approx Kt^n$, where K is a function of temperature, t is the annealing period, and $0 \leq n \leq 1/2$ is the *grain growth exponent*. Prediction of the parameters K and n is a problem more difficult than, for example, the kinetic theory of gases, **"because crystal grains are not independent and separate particles, but connected polyhedra. When one grain changes shape and size, the neighbors of necessity change too." Such an "instability" at a grain corner is eventually spread throughout the array of grains** (Cahn 1974). **Grain**

boundaries exert "forces" on each other. Dislocations are another type of lattice defect which exert "forces," through strain fields, on each other, and these interactions affect material behavior. Second phase particles and solute atoms affect the grain boundary surface tension; accounting for these effects, in addition to those of grain topology, is a large part of the problem in predicting values for the parameters K and n.

The 7475 Al alloy used to illustrate the results of Chapter Two exhibits "non-ideal" grain growth during annealing, *i.e.* n $<<$ 1/2. The behavior of this alloy during annealing and superplastic deformation is described in the Appendix. Superplastic deformation markedly increases the rate of grain growth in 7475 Al; the grain growth rate is proportional to the strain rate, and the grain growth rate also decreases with increasing strain for a given strain rate; the rate of grain growth increases with increasing deformation temperature in a manner that changes with the temperature. Accounting for the effects of strain, strain rate, and temperature, on the grain size of a superplastically deformed material is a very challenging problem in mechanical metallurgy. The grain growth kinetics developed in Chapter Two for 7475 Al describe its behavior in a "semi-quantitative" fashion.

There are several different ways to model grain growth, and these can be classified as either "statistical" or "mechanistic" (Atkinson 1988). *Statistical theories* describe the behavior of the data, *i.e.* the grain size distribution function. *Mechanistic theories* attempt to account for the microstructural events responsible for the grain boundary migration that is required to produce coarsening; but there is relatively little experimental information available on how grain boundaries move (Gore *et al.* 1989), and these theories might consequently be regarded as somewhat speculative.

Three different "levels of description" have been employed to develop mechanisms for grain boundary migration: **(1)** *atomistic theories* treat migration as resulting from atomic diffusion; **(2)** *defect theories* attribute migration to the movement of "steps," "kinks," "ledges," or "grain boundary dislocations;" **(3)** *topological theories*, *e.g.* Hillert (1965) and Rhines and Craig (1974), result from descriptions of migration without specifically addressing the mechanism of migration.

Chen's (1987) theory of grain growth provides an important bridge between these theories: the topological and defect theories of grain boundary migration are united and combined as a statistical theory through the grain size distribution function.

Hillert's (1965) grain growth "reaction" is a description of the "topological transformations" that produce coarsening. This is the level of description used here. Hillert models the polycrystalline microstructure as a two dimensional froth; the first type of *topological transformation* (T1) is neighbor switching of four cells in the array and the second (T2) is cell disappearance. His grain growth "reaction" involves two T1 reactions and one T2 reaction; the T1 reactions turn a 5-sided cell adjacent to a 7-sided cell first into a 4-sided cell and then into a 3-sided cell, which disappears, and in so doing creates another 5-7 combination; disappearance of the 3-sided cell (T2 reaction) results in coarsening. This "reaction" is (thermally-activated) *cellular dislocation climb* (Cahn and Padawer 1965); the 5-7 combination of cells in a hexagonal array is a *cellular dislocation* (Cahn and Padawer 1965, Morral and Ashby 1974).

Superplastic deformation results in large specimen strain with minimal, or even absent grain strain. Since the grains deform so little they must move over and around each other

like flowing sand. Rachinger (1952-53) also observed a lack of grain deformation in large strain creep tests and attributed specimen strain to <u>microstructural flow</u> from <u>translatory grain motions</u>. Lee (1970) and McLean (1970) proposed the <u>neighbor switching mechanism</u> for these grain motions, and McLean (1970) suggested that neighbor switching would occur <u>sequentially</u>. **Plastic deformation of a crystal lattice via dislocation glide or climb is also a *sequential process*: atomic motion does not occur simultaneously throughout a slip plane but only at dislocation cores.** Cellular dislocation climb is, of course, a sequential processes as well.

Desch (1919) suggests that polycrystalline materials can be modelled as a froth with minimal surface area, *i.e.* an array of cells with Kelvin's (Thomson 1887) tetrakaidecahedron of minimum area as the unit, Figure 2d. Throughout this work it is assumed that this type of cell, and its two-dimensional analog the hexagon, is the appropriate model for the grains in a polycrystalline material; but other cell types are possible (Desch 1919, Weaire, Pittet and Hutzler 1993).

Morral and Ashby (1974) note that the "dual" structure for a space-filling array of tetrakaidecahedrons is a body-centered cubic lattice.[1] Similarly the "dual" for a hexagonal array is a lattice with six*fold* symmetry (like the close-packed planes in the face-centered cubic and hexagonal close-packed crystals). Cellular dislocations and disclinations are topological defects associated with *irregular* cells, *i.e.* those which depart from the regular

---

[1]: The tetrakaidecahedron is also the first Brillouin zone of the face-centered cubic crystal, or the Wigner-Seitz cell for its reciprocal lattice (Ashcroft and Mermin 1976).

tetrakaidecahedron (three dimensional) or hexagonal (two dimensional) shape. For example, isolated 5- and 7-sided cells are disclinations in a (two dimensional) hexagonal array, while if these cells are adjacent, then the 5-7 combination is a cellular dislocation. A pair of two 5-7 combinations, one "negative" the other "positive," is analogous to a cellular dislocation loop in a three dimensional froth.

Neighbor switching produces translatory cell motion, and corresponds to cellular dislocation glide if the switching cells are at a dislocation core. Morral and Ashby (1974) therefore suggest that superplastic flow can be modelled as cellular dislocation glide (a sequential process) in a froth. An equivalent model results by considering the plastic deformation of the "dual" lattice to the froth. Sato *et al.* (1990) suggest that stress-driven cellular dislocation climb can model both deformation-enhanced grain growth as well as the microstructural flow.

A "complete" *theory of superplastic deformation* would predict the magnitude of the strain rate and account for the non-Newtonian viscosity of the material, as well as its microstructural evolution, *e.g.* deformation-enhanced grain growth and change in crystallographic texture. The fact that a constitutive relationship predicting a single strain rate sensitivity and not accounting for deformation-enhanced grain growth is an inadequate description of superplasticity was recognized by Ashby and Verrall (1973).

Grains accumulate along the loading axis while the grain density decreases along the transverse directions during superplastic flow; if there is simultaneous grain growth then the grain density decreases with strain as well. Most theories of superplasticity consider the behavior of one group of grains representing an "average"

of the behavior for the aggregate, predict only a single strain rate sensitivity, and do not account for microstructural change. Morral and Ashby (1974) suggest that non-Newtonian behavior results if deformation of an aggregate of grains is inhomogeneous, as is the case for crystal plasticity from dislocation glide and/or climb. *Inhomogeneous superplastic deformation* is the relative motion of parts of the grain aggregate. Zelin *et al.* (1994), and Astanin, Kaibyshev and Faizova (1994), observe this type of behavior as *cooperative grain boundary sliding*. Zelin *et al.* suggest that it must be considered in determining the strain rate sensitivity of the material. **Astanin *et al.* suggest that the number of "deformation bands" is strain rate dependent: at low strain rates a small stress induces deformation in widely spaced "deformation bands," higher strain rates require larger stresses to move more "deformation bands."** Sherwood and Hamilton's (1994) presentation of Morral and Ashby's (1974) theory is consistent with the work of Zelin *et al.* and Astanin *et al.*.

The focus of this work is on describing the microstructural evolution of superplastic flow. A consensus on what type of descriptive methodology is appropriate for a theory of superplastic flow has been somewhat lacking, but may be emerging. I am following Morral and Ashby (1974) by modelling a polycrystalline microstructure as a regular cellular array with cellular dislocations and disclinations, which has a "dual" lattice with corresponding lattice defects (dislocations and disclinations).

Polycrystalline microstructures consist of grains with many different shapes and sizes. This microstructure is modelled with a regular array of cells containing cellular dislocations and disclinations. These defects redistribute cell boundaries in the array, *e.g.* a "dislocation deformation" and the "compatibility theory of dislocations." "Conventional" creep results in grain plasticity without translatory grain motion, except perhaps for large strains (Rachinger 1952-53, Beeré 1978); this is modelled by cell stretching, or "elastic" deformation of the "dual" lattice. Superplasticity results from the relative motion of neighboring grains (Lee 1970, McLean 1970, Ashby and Verrall 1973), or groups of grains (Edington *et al.* 1976, Zelin *et al.* 1994, Astanin *et al.* 1994). Superplastic deformation is described by the flow of a cellular array: cells suffer relative translatory motions (cellular dislocation glide, or dislocation glide in the "dual" lattice) and relative rotational motions (disclination movement). The strain rate is proportional to the mobile cellular dislocation density (Morral and Ashby 1974). Deformation-enhanced grain growth is modelled by cellular dislocation climb (Sato *et al.* 1990, Sherwood and Hamilton 1991,1994). The grain growth rate is also proportional to the cellular dislocation density (Morral and Ashby 1974).

The dislocation density is the mathematical "dual" to the "torsion tensor," which is the "Burgers vector per unit area" (Nabarro 1967).

A cellular dislocation glides at a rate $v = MbT$, where M is its mobility, b its Burgers vector, which is approximately the average cell size d, and T is the shear stress on the "slip

plane" (Morral and Ashby 1974). This rate is $v = \partial \rho / \partial t$, where ρ gives the position of the defect at time t. The mobility M is a function of cell size; this function is determined by assuming an accommodation mechanism for neighbor switching. The term $b\tau$ is the Peach-Koehler (1950) "force" on unit dislocation length, $F_t = b\tau$; thus, $v = MF_t$.

The cellular dislocation density, ρ, is proportional to the number of cells per unit volume ($\propto 1/d^3$) and the total length of the dislocation lines in the array ($\propto X_d d$, X_d denoting the fraction of cells in the array located at dislocation cores); thus, $\rho \propto X_d/d^2$. Dislocation motion under the direction of stress produces plastic deformation with the *shear rate* being given by the *Orowan equation*, $\Delta\gamma/\Delta t = \rho b v$, which I develop as a "Pfaffian" (exterior calculus terminology), $\Delta\gamma/\Delta t$, in Chapter Two. Thus, $\Delta\gamma/\Delta t = \rho b^2 M\tau \approx X_d M\tau$; if $X_d M = $ *constant* then Newtonian viscosity is predicted; if $X_d M$ is a function of strain (*e.g.* from cell coarsening) and/or stress then non-Newtonian viscosity is predicted. These assertions are examined in Chapter Two by modelling the behavior of a single phase Sn-1% Bi alloy and a quasi-single phase 7475 Al alloy.

If the imposed strain rate is larger than that given by the Orowan equation, either because the temperature is too low so that M is small or X_d is small, then cells will stretch instead of flowing with the strain rate, non-Newtonian viscosity resulting again, here as a model for high strain rate creep (Region III superplasticity or higher). If the imposed strain rate produces a stress too small to move the dislocations, *i.e.* $v = Mb\tau \approx 0$ because τ is less than some "threshold stress," then non-Newtonian viscosity results again, as a model for

Region I superplasticity (Hamilton 1994, personal communication).

Morral and Ashby's (1974) theory therefore appears to be capable of predicting the transition: diffusional creep → Region I → Region II → Region III.

The coarsening rate from thermally-activated cellular dislocation climb is $\partial d/\partial t = \frac{1}{3}X_d[M_B(2\Gamma/d)]$ if cell boundaries are unimpeded; here, X_d is the fraction of defects engaged in Hillert's (1965) grain growth "reaction," M_B is the boundary mobility and Γ the surface tension. Integration of this relationship results in a prediction of *"ideal grain growth"*, n = 1/2, if $X_d = constant$, and "non-ideal" grain growth if X_d decreases with increasing t or d (Morral and Ashby 1974). If the cellular dislocation mobility is inhibited by "particles," then $\partial d/\partial t \approx \frac{1}{3}X_d[M_B(2\Gamma/d)]|1 - d/d_c|^2$, d_c denoting the cell size at which dislocations are immobilized by the particles (Hillert 1965), a model for grain growth in quasi-single phase materials. This expression is used to model "non-ideal" grain growth and deformation-enhanced grain growth in 7475 Al (Sherwood and Hamilton 1994) in Chapter Two.

I develop the theory of dislocations with tensor calculus based on differential forms, *exterior calculus*.[2] Birss (1983) makes the case for application of this mathematics to crystallography, suggested earlier in a casual manner by Misner *et al.* (1973), and actually

---

[2]: See Munroe (1963) for an introductory treatment, and also Misner and Wheeler (1957); Misner *et al.* (1973), Marsden and Hughes (1983), Wald (1984), Burke (1985), Schouten (1989), and Wasserman (1992) provide more advanced versions of the subject; Schouten (1954), Schouten and Kulk (1969), Choquet-Bruhat, DeWitt-Morette and Dillard-Bleick (1982), Edelen (1985), and Bryant *et al.* (1991) give the most advanced treatments.

used by Bilby (1960) and his coworkers (Bilby, Bullough and Smith 1955, Bilby and Smith 1956, and Bilby *et al.* 1958), Kondo (1964), and Nabarro (1967). It has recently been used by Dereli and Vercin (1987), and Kröner and Lagoudas (1992), to describe defects in amorphous materials. de Wit (1981) introduces the "component version" of exterior calculus for dislocation theory. I introduce the "geometric version," after Misner *et al.* (1973).

> *"In a crystal the dislocations are necessarily discrete, so that the plastic strain is concentrated at the dislocation cores and the material between them is strained elastically. However, to make the connection with continuum theory we must visualize the lattice constant to approach zero dimension and the dislocation density to become continuous throughout the crystal, thus eliminating all elastic strain."* From de Wit (1981).

I reserve the term "plastic strain" for the permanent deformation of the <u>material</u> that transpires from the motion of defects through the lattice. I use "dislocation deformation" for de Wit's (1981) (Nabarro (1967) and Kosevich (1979) too) "plastic deformation concentrated at dislocation cores." See Vitek (1992) for a recent description of the dislocation core.

The grains in a polycrystalline microstructure undergoing superplastic deformation have three translational degrees of freedom and three rotational degrees of freedom. Translational grain motion is represented by cellular dislocation glide while rotational grain motion is represented by cellular disclination motion. These two types of motion are not sufficient to fully describe superplastic flow because, in order to maintain a continuous grain

24

structure, it is likely that some grains will also have to be deformed (stretch) to maintain contact with their neighbors. This grain stretching provides three more degrees of freedom to each grain. Grains may also be periodically eliminated from the microstructure, resulting in an increase in the average grain size (grain growth), or produced in the microstructure, resulting in a decrease in the average grain size (dynamic recrystallization). Considering only grain elimination then, the total number of degrees of freedom allotted to each grain is ten.

There are no endorsements here of particular accommodation mechanisms for translatory grain motions, analogously perhaps, to this thought from Misner *et al.* (1973):

"In a mixed solid there are hundreds of distinct bonds, all of which contribute to the elastic constants; some of them arise from Van der Waal's forces, some from ionic coupling, some from homopolar linkage; they have the greatest variety of strengths; but all have their origin in something so fantastically simple as a system of positively and negatively charged masses moving in accordance with the laws of quantum mechanics. In no way was it required or right to meet each complication of the chemistry and physics of a myriad of bonds with a corresponding complication of principle. By going to a level of analysis deeper than bond strengths, one had emerged into a world of light, where nothing but simplicity and unity was to be seen."

I assume throughout this work that cellular dislocations are entirely analogous to their crystalline counter parts, following Morral and Ashby (1974) in this regard:

25

"Many of the properties of dislocations in crystals should apply to dislocations in cellular structures as well. For example, the Burgers vector conservation rules must be obeyed when dislocations combine or meet at nodes. In addition, cellular dislocations can multiply, cross slip, intersect one another, interact with pores and inclusions, and combine to form boundaries between perfect cellular structures."

A *crystal* is an array of atoms packed into a single pattern (*lattice*) that is repeated throughout space; they are *anisotropic substances* since material properties are dependent on crystallographic direction. A unique volume can be assigned to the atoms in a crystal lattice, the atomic volume, Ω_0. This volume has three characteristic vectors $\mathbf{A}_M$ which serve to define a crystallographic coordinate system X^M. Each atom (or atomic volume element) is assigned to a point $\wp_0$ of this coordinate system. When a crystal is deformed the volumes at each $\wp_0(X^M)$ are carried to volumes Ω at points $\wp(x^m)$ characterized by three vectors $\mathbf{a}_m$, where x^m is the crystallographic coordinate system of the deformed lattice. The coordinates X^M and x^m for each atom before and after deformation are generally in different regions of space, and even though the vectors $\mathbf{A}_M$ and $\mathbf{a}_m$ point to the same neighboring atoms in the lattice, these vectors will generally point to different directions in space. So to describe the deformation of a crystal, both atomic locations and characteristic directions need to be assessed; the mathematical model for this is a *Cosserat Continuum* (Truesdell and Toupin 1960).